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(21) International Application Number: PCT/AU95/00057 (22) International Filing Date: 8 February 1995 (08.02.95) (30) Priority Data: PM 3835 11 February 1994 (11.02.94) AU (71) Applicant (for all designated States except US): INCITEC LTD. [AU/AU]; Paringa Road, Gibson Island, Murarrie, 4172 (AU). (72) Inventor; and (75) Inventor/Applicant (for US only): BROWN, Anthony, Martin [AU/AU]; 9 Doheny Street, Mt Gravatt, QLD 4122 (AU). (74) Agent: GRANT ADAMS & COMPANY; Patent & Trade Mark Attorneys, 215 Adelaide Street, G.P.O. Box 1413, Brisbane, QLD 4000 (AU).		(81) Designated States: AU, CA, CN, US. Published <i>With international search report.</i>
(54) Title: GRANULAR UREA (57) Abstract A method for producing free-flowing, non-caking granular urea of at least 2.00 mm particle size and of at least 1.5 kg crush strength. The granulation is undertaken in the presence of a conditioning agent and a granulating aid. The conditioning agent is a divalent metal oxide such as calcium, magnesium or zinc oxide. The granulating aid is a trivalent metal salt such as aluminium or ferric sulphate.		

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TITLE: GRANULAR UREA

FIELD OF THE INVENTION

The present invention relates to a process for the production of granular urea, and to the granular urea produced by that process. This invention is particularly useful for the production of granular urea of particle size greater than 2 mm, of a greater than 1.5 kg crush test and which remains free-flowing and substantially free from caking during handling and storage.

BACKGROUND OF THE INVENTION

Urea (also known as carbamide or carbonyldiamide) has a number of important applications, including use as a diuretic, as a partial source of dietary nitrogen in ruminants, in ammoniated dentifrices, in the paper industry to soften cellulose, and in fertilizers. Perhaps the chief among these uses is in agricultural fertilization. In this capacity, urea provides an easily available major source of nitrogen, which is a critical crop nutrient.

Urea is often not used as a fertilizer by itself, but rather in combination with other vital plant nutrients. Therefore, in commercial use, urea must often be blended with granular fertilizers to produce a balanced fertilizer blend.

Traditionally, fertilizer grade urea has been produced using prilling methods where molten urea is sprayed through a rose at the top of a prilling tower and the resultant droplets of urea solidify as they fall through

a cooling stream of air.

This technology has several limitations:

1. The size of the urea particle is limited to under 2.00 mm diameter. Hence, the prill does not blend well with other types of fertilizers of larger particle size in mixtures blended to suit crop needs. This results in segregation of the mixture during handling processes and maldistribution of the fertilizer on crops.
2. The prilling process creates significant dust (that is, material under 1.00 mm in diameter). This dust, which is usually in excess of 1.0% of manufacture and can be as high as 10%, causes windage losses on application, segregates badly on handling and encourages rapid moisture absorption and caking of the fertilizer on storage.
3. The prills are usually fairly weak in structure - having a crush strength usually of less than 1.5 kg - and tend to break down during handling and storage. This further contributes to dust problems.

For these reasons, in recent years, plants which are capable of producing a granular urea of larger particle size and a harder particle have come into production.

- 25 There are several methods of producing granular urea including NSM fluid bed granulation, spherodisers, pan granulation techniques and spouted bed granulators. These plants all produce a granule from a molten urea feed containing less than 10% water in the melt.

Processes have also been developed which can take a part prill feed and coat and solidify a urea melt onto the prills thus fattening the prills. Examples are the TVA falling curtain granulation techniques and Kaltenbach granulation plants.

However, the high capital cost of all these alternative granulation routes has meant only a slow introduction of the technology as the traditional prilling techniques are much lower in operating costs. In addition, if the urea is used in a straight application and is not blended, then a good quality prill is more uniform and free flowing than a granule.

As will be appreciated from the foregoing discussion, there is a need for a process which can granulate urea fines to a uniform but larger particle size of a superior hardness to produce granular urea which can readily be used as, inter alia, a fertilizer, by itself or, if necessary, in combination with other components.

One possible solution may have been the adaption of the process described in Australian Patent No. 627438 wherein another nitrogen-containing fertilizer component - ammonium sulphate - is granulated in the presence of a trivalent metal salt, leading to a hard, free-flowing, non-caking granule.

It was anticipated that a similar process applied to urea may produce suitable granules of urea. Unfortunately, tests proved that simply replacing urea for the ammonium sulphate was not satisfactory as the resultant combination was of high solubility and low critical humidity leading to a "wet" or "soft" and sticky product which would lead not only to intolerable

blockages of the granulation plant, but also to an unacceptable product for storage and handling.

However, it has now been established that if a conditioning agent is used in conjunction with the
5 granulating aid, this particular problem can be overcome.

SUMMARY OF THE INVENTION

According to the present invention, granulation can be achieved by feeding a stream of urea fines - or other
10 suitable urea particulates - and a conditioning agent to a granulator where granulation is then carried out in the presence of a granulating aid. The resulting product can then, of course, be dried, screened and cooled by way of any suitable available techniques.

15 The conditioning agent is preferably divalent metal oxide. Preferred divalent oxides include calcium, magnesium and zinc oxides.

The granulating aid is preferably a metal salt or derivative thereof. Preferred metal salts are mono-,
20 di- or trivalent cation salts or derivatives thereof. The more preferred trivalent cations are aluminium and ferric ions. The most preferred metal salts include aluminium and ferric oxides and sulphates and hydrates thereof.

25 Any conventional granulator may be used, including rotary granulators, pugmills, drums and blungers.

Without being bound by theory, it is proposed that the conditioning agent reacts with the granulating aid to

prevent the creation of a highly soluble double salt, and it also absorbs moisture thus inhibiting softening of the product and subsequent caking. This is an important property of the conditioning agent because, as
5 mentioned above, wet or soft product in a granulating plant leads to unacceptable blockages of the granulation plant. In combination with the granulating aid, the conditioning agent results in a larger size, hard, free flowing granule of urea.

10

DESCRIPTION OF PREFERRED EMBODIMENTS

The present invention may be further appreciated from the following Examples with reference to the accompanying drawing. It is to be understood that these Examples are merely illustrative and in no way define
15 or limit the scope of the present invention, which extends to any and all compositions, means, and methods suited for practice of the process according to the present invention, as well as to any and all products made thereby.

20 General Example

The drawing represents the process diagram for a typical NPK granulation plant. A urea fines feed (7) is fed into the granulation drum or blunger together with a modulated stream of divalent metal oxide (4) at the
25 appropriate composition.

Steam (3) is used to sparge under the bed and assist in granulation.

A solution or slurry of a trivalent metal (2) such as aluminium or iron, eg. alum sulfate, is sprayed into the

granulator onto the bed.

A spray or scrubber liquor (6) may in some cases be used to add more moisture to the granulation.

A recycled stream of fines may also be fed back to the
5 granulation equipment (11).

The granulated wet material is then fed to a drier where heat is supplied usually concurrently and moisture is dried off.

The dried material (12) is then fed to screens where the
10 product size is separated and either sent direct to storage (14) or cooled and sent to storage. The undersize is recycled and the oversize is crushed and also recycled to the granulator.

The air streams from the granulator and drier are
15 scrubbed, usually in venturii scrubbers (10) and (16).

The scrubber liquor is recirculated to the scrubbers and a side stream is usually fed to the granulator to supplement the water balance in the granulator. In some cases, this does not occur and the scrubber liquor is
20 accumulated for later disposal.

The process of the present invention is conducted in such a plant at a typical production rate of 10 to 20 tonnes per hour. When the granulating aid is an aluminium salt, the amount of the aluminium salt added
25 to the slurry is sufficient to produce urea granules having an aluminium content of between 0.05% and 1.06% by weight; preferably of between about 0.15% and 1.06% by weight, and most preferably of between about 0.20%

and 0.60% by weight.

Specific Example

Following the general example described above, undersize urea fines obtained from prilled urea are fed into a traditional NPK drum granulation plant at 10 tonnes/hr together with 300 kg/hr of ground magnesium oxide powder, using a 1500 kg/hr steam sparge together with 0.2 l/sec 50% alum sulphate solution. The overall moisture content is increased using scrubber liquor sprayed into the granulator. After granulation, the product is dried to produce urea granules containing 0.3% aluminium and 0.7% magnesium by weight; 90% of the product exceeding 2.00 mm in size; and of 4.0 kg crush strength.

Granules produced according to the present invention were subjected to a caking test. Bag tests were conducted using the TVA bag test method described in Bulletin Y-147 and slightly adapted in that material was placed in 40 kg good quality fertilizer bags and stacked four layers or 1 tonne per pallet and then stacked four pallets high for one week, one month and three month trials. The bags were opened and inspected at those times and the degree of set and hardness of set were determined. This material consistently was only partially set and light finger pressure broke the lumps. If a bag was dropped from waist height the light set completely shattered.

After several weeks of bulk storage, the product remained so lightly set that any disturbance rendered it free-flowing.

Granulated urea produced according to the present invention is substantially harder than prilled urea known to the prior art and equal to granular urea produced by other processes.

- 5 This hardness of the granules was measured with a commercial compression tester, a Chatillon compression tester. At least 25 granules from a given product run were tested individually, and the average of these measurements was taken as the hardness of the product
10 run from which the tested granules were taken. The granules were placed, one at a time, on a flat surface provided on the compression tester. Pressure was applied to each granule using a flat-end rod attached to the compression tester, and a gauge mounted in the
15 compression tester measured the pressure required to fracture the granule. The urea granules produced according to the process of the present invention generally possessed a hardness in the range of from 2.5 to 4.0 kgs. This is to be compared with the prior art
20 where prilled urea has typical hardness values of 1.5 kg or less.

- Granulated urea produced according to the present invention remains free-flowing and does not consolidate or set to a hard mass upon being allowed to stand in
25 large piles during storage. In addition, the granules are of a size (at least 70%, but usually excess of 90%, of the product exceeds 2.00 mm) and hardness (at least greater than 1.5 kg crush strength, usually approximately 4.0 kg) superior to the prilled urea known
30 from the aforementioned prior art.

Due to the superior size and hardness of these granules, urea produced according to the present invention

experiences minimal breakdown into undesirable small fragments during cooling, storage, handling, blending, shipping and spreading.

Moreover, use of granular urea produced according to the present invention, either as a fertilizer per se or as an ingredient in fertilizer blends, produces exceptionally uniform results. This follows from the fact that the mechanics of spreading fertilizer are improved by use of a physically more uniform product, resulting in more uniform spreading. In addition, when fertilizer blends are used, blends using granular urea produced according to the present invention will remain uniformly blended, rather than tending to layer out by component by the time the end use is reached as is often the case with prior art blends.

Yet another advantage is that the divalent metal oxide can not only function as a conditioning agent but also as a trace element additive in certain fertilizer blends.

Further, the present invention allows urea fines generated from traditional prilling plants to be granulated to a useful end product using traditional granulation plants. Thus, the significant capital cost of establishing a specialised urea granulation plant is avoided entirely.

Those skilled in the art will appreciate that the above embodiments are given by way of exemplification of the invention only, and that changes may be made to the details set out therein without departing from the scope of the invention as defined in the following claims.

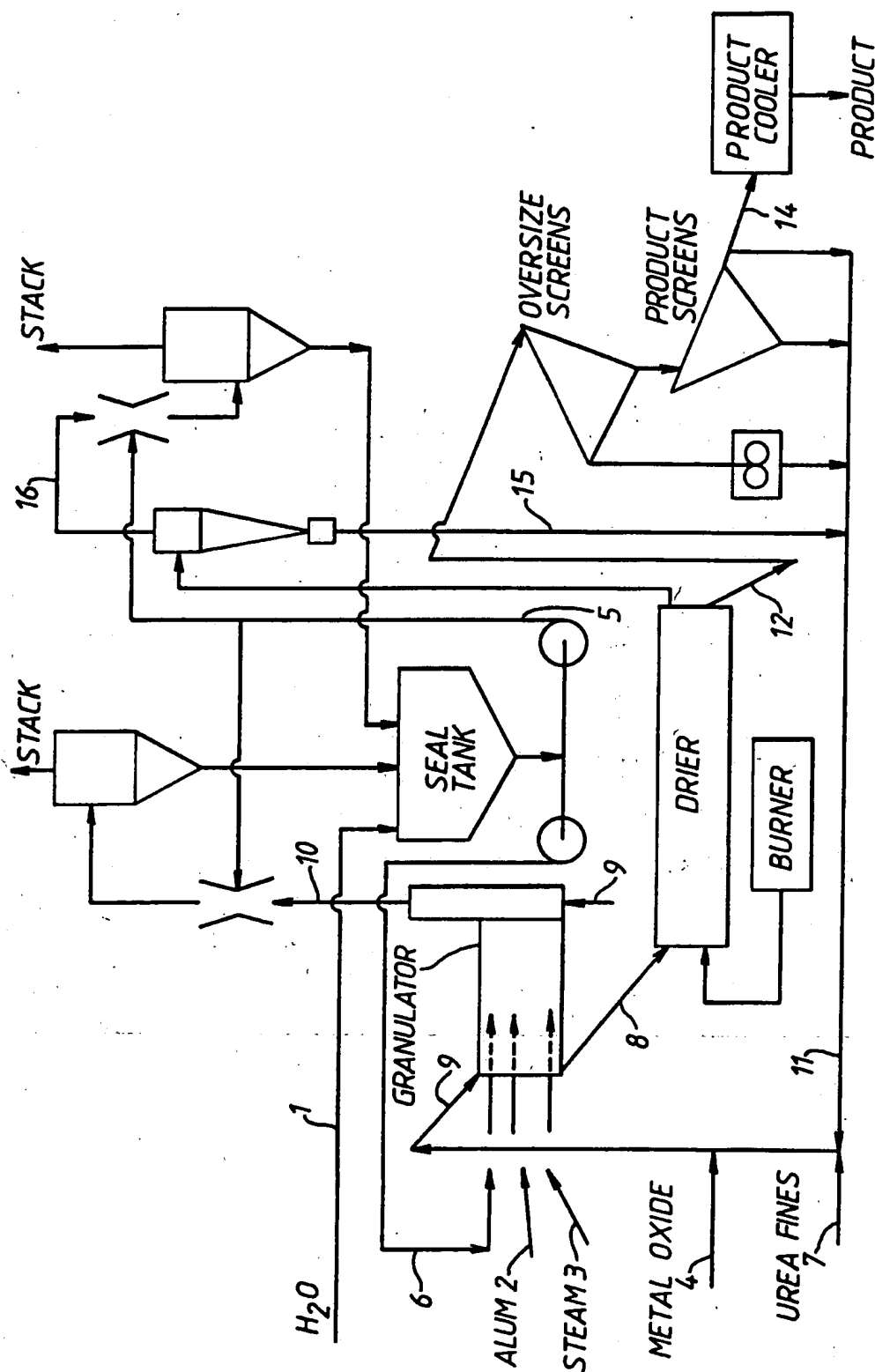
CLAIMS:

1. A method for producing granular urea, said method comprising:
 - a) feeding urea to be granulated and a conditioning agent to a granulator; and
 - b) granulating the resultant mixture in the presence of a granulating aid.
2. A method as defined in Claim 1, wherein said conditioning agent is a divalent metal oxide.
- 10 3. A method as defined in Claim 2, wherein said metal oxide is selected from the group consisting of calcium, magnesium and zinc oxides.
4. A method as defined in any one of Claims 1 to 3, wherein said granulating aid is a metal salt or hydrate thereof.
- 15 5. A method as defined in Claim 4, wherein said metal salt is an aluminium or ferric salt or hydrate thereof.
6. A method as defined in Claim 5, wherein said aluminium or ferric salt is aluminium or ferric sulphate.
- 20 7. A method as defined in any one of Claims 1 to 6, wherein said granular urea is at least 2.00 mm in size.
- 25 8. A method as defined in any one of Claims 1 to 7, wherein said granular urea is at least of greater

than 1.5 kg crush strength.

9. A method as defined in Claim 8, wherein said granular urea is at least of 4.0 kg crush strength.
10. Granular urea prepared by a method as defined in
5 any one of Claims 1 to 9.
11. Granular urea which is free-flowing and substantially non-caking of at least 2.00 mm particle size and of at least 1.5 kg crush strength.

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU 95/00057

A. CLASSIFICATION OF SUBJECT MATTER Int. Cl. ⁶ B01J 2/30; C05C 9/00, 7/02 According to International Patent Classification (IPC) or to both national classification and IPC				
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) B01J 2/30; C05C 7/02, 9/00; C07C 273/02, 126/00, 127/00 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base, and where practicable, search terms used) DIALOG: UREA, GRANULE, SPHERE, PRILL				
C. DOCUMENTS CONSIDERED TO BE RELEVANT				
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.		
X	AU 42647/78A1 (FISONS LTD) 28 June 1979. Page 3 Lines 5-10, Page 4 Line 20 to Page 9 Line 8, Examples 1-21, Claims 1-7.	1-7 and 10		
X	SU 1224298A1 (BELORUSS KIROV TECHN INS) 15 April 1986.	1-8, 10 and 11		
X	SU 1289865A1 (MEEROYSKAYA V D) 15 February 1987.	1-6, 8-10		
X	SU 1337377A1 (TEPLITSKII Y.A.S.) 15 September 1987.	1-6		
X	SU 1421728A1 (YUNUSOV D.K.H.) 7 September 1988.	1-8		
<div style="display: flex; justify-content: space-between;"> <div style="text-align: left;"> <input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. </div> <div style="text-align: right;"> <input checked="" type="checkbox"/> See patent family annex. </div> </div>				
<table style="width: 100%; border: none;"> <tr> <td style="width: 50%; vertical-align: top;"> <p>* Special categories of cited documents :</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </td> <td style="width: 50%; vertical-align: top;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle of theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&" document member of the same patent family</p> </td> </tr> </table>			<p>* Special categories of cited documents :</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p>	<p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle of theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&" document member of the same patent family</p>
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Date of the actual completion of the international search 19 May 1995		Date of mailing of the international search report 30 May 1995 (30.05.95)		
Name and mailing address of the ISA/AU AUSTRALIAN INDUSTRIAL PROPERTY ORGANISATION PO BOX 200 WODEN ACT 2606 AUSTRALIA Facsimile No. 06 2853929		Authorized officer DEBORAH LALLY Telephone No. (06) 2832533		

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU 95/00057

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate of the relevant passages	Relevant to Claim No.
X Y	GB 2077722A1 (COMPAGNIE NEERLANDAISE DE L'AZOTE S.A.) <u>23 December 1981</u> . Page 1 Line 49 to Page 2 Line 9, Tables A, B and C.	<u>11</u> 1-10
X Y	US 4500336A1 (COMPAGNIE NEERLANDAISE DE L'AZOTE) <u>19 February 1985</u> . Column 2 Line 64 to Column 3 Line 14, Tables A, B and C.	<u>10</u> 1-10
X Y	GB 1311065A1 (BADISCHE ANILIN & SODAFABRIK AKTIENGESELLSCHAFT) <u>21 March 1973</u> . Page 1 Lines 54-77, Table.	<u>11</u> 1-8 and 10
X Y	US 4219589A (COMPAGNIE NEERLANDAISE DE L'AZOTE) <u>26 August 1980</u> . Column 1 Lines 49-62, Column 2 Lines 35-50, Table A and D.	<u>11</u> 1-8 and 10
X Y	US 4478632A (COMPAGNIE NEERLANDAISE DE L'AZOTE) <u>23 October 1982</u> . Table B.	<u>11</u> 1-8 and 10
X Y	US 4494976A (UNION OIL COMPANY OF CALIFORNIA) <u>22 January 1985</u> . Examples 2 and 4.	<u>11</u> 1-8 and 10
X Y	US 4701353A (UNIE VAN KUNSTMESTFABRICKEN B.V.) <u>20 October 1987</u> . Column 6 Lines 18-21, Example 3.	<u>11</u> 1-6 and 10
Y	AU 69267/81A1 (UNIE VAN KUNSTMESTFABRICKEN B.V.) <u>15 October 1981</u> . Page 2 Line 11-17, Claim 5.	1-6 and 10
Y	AU 64168/86A1 (NEDERLANSE STICKSTOFFE MAATSCHAPPIJ B.V.) <u>30 April 1987</u> . Page 4 Lines 11-24, Example 2, Table at Page 11, Page 7 Lines 3-26.	1-6 and 10
Y	US 3981713A (MISSISSIPPI CHEMICAL CORPORATION) <u>21 September 1976</u> . Column 2 Lines 32-57, Column 4 Lines 34-48.	1-6 and 10
Y	SU 542750A1 (AS UZB CHEM INST) <u>15 April 1977</u> .	1-5
Y	SU 1010046A1 (BELORUSS KIROV TECHN INS) <u>7 April 1983</u> .	1-3
A	AU 26011/88A1 (RETEC LTD) <u>18 May 1989</u> .	1-11

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU 95/00057**Box I Observations where certain claims were found unsearchable (Continuation of Item 1 of first sheet)**

This international search report has not established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claim Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of Item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU 95/00057

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member			
AU	42647/78	BE	872917	BR	7808419
		DE	2855036	DE	2855036
		DK	5312/78	FI	783878
		FR	2412512	GB	2010797
		IT	1160398	JP	54091471
		NO	784334	SE	7812996
		SU	1289865	SU	1289865
GB	2077722	SU	1224298	SU	1337377
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AU	64168/86	BR	8605068	CA	1281911	CN	86106555
		EP	223276	FI	864127	IN	166616
		JP	62091484	NL	8502838	NO	864144
		NZ	217900	SU	1671162	US	4943308
		ZA	8607665				
AU	26011/88	EP	386043	WO	890518		
<p>END OF ANNEX</p>							